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INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT Article 36 and Rule 70)

REC'D 29 DEC 2004

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| Applicant's or agent's file reference P15256PC00 | | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416) | |
| International application No. PCT/IB 03/04103 | International filing date (day/month/year) 15.09.2003 ✓ | Priority date (day/month/year) 17.09.2002 ✓ | |
| International Patent Classification (IPC) or both national classification and IPC C22B3/00 | | | |
| Applicant CRUNDWELL, Frank Kenneth ✓ | | | |
| <p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 11 sheets. ✓</p> | | | |
| <p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the opinion</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input type="checkbox"/> Certain observations on the international application</p> | | | |
| Date of submission of the demand 08.04.2004 ✓ | | Date of completion of this report 28.12.2004 | |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | | Authorized Officer Bjoerk, P Telephone No. +49 89 2399-8452  | |

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/IB 03/04103

I. Basis of the report

1. With regard to the elements of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, Pages

2-7, 9-51 as originally filed
1, 8 filed with telefax on 27.08.2004

Claims, Numbers

1-58 filed with telefax on 27.08.2004

Drawings, Sheets

1/31-31/31 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
☐ the language of publication of the international application (under Rule 48.3(b)).
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
☐ filed together with the international application in computer readable form.
☐ furnished subsequently to this Authority in written form.
☐ furnished subsequently to this Authority in computer readable form.
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished..
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

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5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | |
|-------------------------------|-------------|------|
| Novelty (N) | Yes: Claims | 1-58 |
| | No: Claims | |
| Inventive step (IS) | Yes: Claims | 1-58 |
| | No: Claims | |
| Industrial applicability (IA) | Yes: Claims | 1-58 |
| | No: Claims | |

2. Citations and explanations

see separate sheet

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EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/IB 03/04103

Re Item V

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. The present application relates to a method of controlling a heap leach process by controlling the irrigation rate as a function of at least one of an aeration rate, an advection measurement and a temperature measurement as well as by controlling an aeration rate as a function of a determination of the oxidation rate of material within the heap.

The application further relates to preliminary method steps of increasing the temperature of a heap (claim 29), of introduction and activation of microorganisms in order to avoid a "skin-plugging" effect (claims 34, 39 and 46).

It is noted that the subject matter of claims 34 to 50 is covered in the parallel application IB03/04186 by the present applicant.

2. Reference is made to the following documents:

D1: US-A-6 110 253

D2: BRIERLEY C L: 'Bacterial succession in bioheap leaching'
HYDROMETALLURGY, ELSEVIER SCIENTIFIC PUBLISHING CY.
AMSTERDAM, NL, vol. 59, no. 2-3, February 2001 (2001-02), pages 249-255, ISSN: 0304-386X

D3: MACLEOD F A ET AL: 'PLUGGING OF A MODEL ROCK SYSTEM BY USING STARVED BACTERIA' APPLIED AND ENVIRONMENTAL MICROBIOLOGY, WASHINGTON, DC, US, vol. 54, no. 6, June 1988 (1988-06), pages 1365-1372, ISSN: 0099-2240

D1 discloses a high temperature heap bioleaching process of chalcopyrite bearing ore (abstract). The irrigation rate is said to depend on a number of factors. In particular, when the temperature of the heap is sufficiently raised, biooxidation of the chalcopyrite will be more rapid and the irrigation rate of process leach solution will also need to be higher in order to lower the pH and remove copper that has been dissolved (col.15, l.29-53). The heap is equipped with one or more temperature monitoring devices such as thermocouples (col.11, l.10-15).

3. The process of claim 1 differs from that described in D1 through the controlling of

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an aeration rate of the heap as a function of the oxidation rate.

Such a feature leads to an optimized aeration rate according to the description on page 27, lines 19-25.

Neither D1 nor any of the remaining prior art cited hints at controlling an aeration rate of the heap as a function of the oxidation rate. D1 appears to only measure the oxygen level in the heap.

Consequently, the subject matter of claim 1 and of its dependent claims 2 to 54 fulfills the requirements of Art.33(2) and (3) PCT.

4. Claims 55 to 58 refer to the description and the figures and their subject matter does therefore not comply with Rule 6.2(a) PCT.

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HEAP LEACH PROCESS

10 FIELD OF THE INVENTION

This invention relates to heap oxidation and leaching for the recovery of metals from ore, including but not limited to bio-assisted heap oxidation.

15 BACKGROUND TO THE INVENTION

Bio-assisted heap leaching for recovery of base metals is only carried out commercially on secondary copper sulphide ores. Recent work in Australia has seen the introduction of heap leaching for recovery of nickel from nickel sulphide ores on a semi-commercial test basis ⁽¹⁾. Bio-assisted heap oxidation of refractory gold ores is also used as a pre-treatment process for recovery of gold from such ores.

Typically the secondary copper sulphide heaps operate at temperatures in the range of 10°C to 25°C and rely on the exothermic oxidation of secondary copper sulphide minerals to keep the temperature of the heap above ambient conditions. The relatively low temperature limits the rate of sulphide mineral oxidation that can be achieved. Additionally, chalcopyrite ores cannot be leached at these low temperatures because chalcopyrite is generally considered to be refractory to leaching at such temperatures.

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irrigation rate per unit area; and the phrase "instantaneous aeration rate" means the instantaneous gas flow rate applied over any time period shorter than the total duration of the leach cycle expressed as instantaneous hourly aeration rate per unit area.

- The terms irrigation rate and aeration rate refer to the instantaneous irrigation rate and the instantaneous aeration rate respectively, unless otherwise stated.
- The term "heap leaching" means leaching of ore in heaps or dumps.
- The term "oxygen utilization of the heap" means the total oxygen consumed within the heap expressed as a percentage of the total oxygen passed through the heap.
- The term advection means the net transfer of energy up or down the heap.

OBJECT OF THE INVENTION

It is an object of this invention to provide a heap leaching process that at least partly alleviates some of the abovementioned problems.

SUMMARY OF THE INVENTION

In accordance with this invention there is provided a method of controlling a heap leach process through controlling an irrigation rate of a heap as a function of at least one of an aeration rate of the heap, a determination of advection at least at one predetermined point in the heap, and a determination of temperature at least at one predetermined point in the heap.

There is further provided for the heap to be aerated by means of natural convection, and for the natural convection to be at least partly induced.

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CLAIMS

1. A method of controlling a heap leach process through
5 controlling an irrigation rate of a heap as a function of at least one of
an aeration rate of the heap, a determination of advection at least at
one predetermined point in the heap, and a determination of
temperature at least at one predetermined point in the heap, and
controlling an aeration rate of the heap as a function of a
10 determination of the oxidation rate of material within the heap.
2. A method as claimed in claim 1, in which aeration of the heap
is by natural convection.
- 15 3. A method as claimed in claim 2 in which the natural convection
is at least partly induced.
4. A method as claimed in claim 1 in which the aeration is forced.
- 20 5. A method as claimed in any one of claims 1 to 4, which
includes determining the advection at or below the heap surface.
6. A method as claimed in claim 5, which includes determining
the advection at a point from 0% to 95% of the heap height below the
25 heap surface.
7. A method as claimed in claim 6, which includes determining
the advection at a point from 1% to 40% of the heap height below the
heap surface.
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8. A method as claimed in claim 7, which includes determining the advection at a point from 2% to 30% of the heap height below the heap surface.

5 9. A method as claimed in claim 1 to 8, which includes controlling the aeration rate to maintain a predetermined oxygen utilization of the heap.

10 10. A method as claimed in claim 9, which includes maintaining the oxygen utilization of the heap in the range of 1% to 99%.

11. A method as claimed in claim 10, which includes maintaining the oxygen utilization of the heap in the range of 15% to 90%.

15 12. A method as claimed in claim 11, which includes maintaining the oxygen utilization of the heap in the range of 20% to 85%.

20 13. A method as claimed in any one of the previous claims, which includes maintaining the average aeration rate and average irrigation rate at a ratio in the range of 0.125:1 to 5:1.

14. A method as claimed in claim 13, which includes maintaining the average aeration rate and average irrigation rate at a ratio in the range of 0.15:1 to 2:1.

25 15. A method as claimed in claim 14, which includes maintaining the average aeration rate and average irrigation rate at a ratio in the range of 0.175:1 and 1.5:1.

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16. A method as claimed in claim 15, which includes maintaining the average aeration rate and average irrigation rate at a ratio of about 0.2:1.

5 17. A method as claimed in any one of the previous claims, which includes maintaining the instantaneous aeration rate and instantaneous irrigation rate at a ratio in the range of 0:1 to 5:1.

10 18. A method as claimed in claim 17, which includes maintaining the instantaneous aeration rate and instantaneous irrigation rate at a ratio in the range of 0:1 to 2:1.

15 19. A method as claimed in claim 18, which includes maintaining the instantaneous aeration rate and instantaneous irrigation rate at a ratio in the range of 0:1 and 1.5:1.

20 20. A method as claimed in claim 19, which includes maintaining the instantaneous aeration rate and instantaneous irrigation rate at a ratio of about 0.2:1.

21. A method as claimed in any one of the previous claims, which includes determining the temperature below the heap surface.

25 22. A method as claimed in claim 21, which includes determining the temperature at a point from 1% to 95% of the heap height below the heap surface.

30 23. A method as claimed in claim 22, which includes determining the temperature at a point from 5% to 50% of the heap height below the heap surface.

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24. A method as claimed in claim 23, which includes determining the temperature at a point from 10% to 30% of the heap height below the heap surface.
- 5 25. A method as claimed in any one of claim 1 to 20, in which the temperature determination includes a determination of the pregnant leach stream temperature.
- 10 26. A method as claimed in any one of claims 1 to 24 in which the oxidation rate of sulphide material is determined as a function of any one or more of determinations of the oxygen content of the heap gas, the pregnant leach stream temperature, the heap temperature, the pregnant leach stream metal content, the pregnant leach stream redox value, the pregnant leach stream oxygen concentration, the
- 15 heap oxygen uptake rate, the heap carbon dioxide uptake rate, simulation based on at least feed composition, sulphide mineral leaching rates, heap geometry, climatic conditions external to the heap, and historical values of previously leached heaps.
- 20 27. A method as claimed in claim 26 in which the pregnant leach stream metal content includes recovered metal content.
28. A method of increasing the temperature of heap of material for heap leaching by:
- 25 a) equipping a support surface for the heap with aeration and drainage equipment;
- b) forming a layer of granular material on the support surface,
- c) installing an irrigation system proximate the operative upper surface of the layer of granular material,
- 30 d) forming a layer of ore on the granular material layer;

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- e) passing a hot solution through the granular layer by means of the irrigation system to heat the granular layer,
- f) blowing ambient air through the aeration equipment of the support surface to react with the layer of ore until the temperature of the ore heap reaches a predetermined take-off point,
- 5 g) at least reducing the hot solution irrigation flow of step e) through the granular layer,
- h) introducing irrigation of the ore layer and adjusting the aeration through the aeration equipment until a predeterminable normal
- 10 optimum heap temperature is reached, and
- i) controlling the heap leaching process according to any one of claims 1 to 27.

29. A method as claimed in claim 28 in which step d) includes
15 inoculation of the layer of ore with suitable microorganisms and at least some acid.

30. A method as claimed in claim 28 or 29 in which the granular
20 layer is formed from crushed rock.

31. A method as claimed in claim 28 to 30 in which the hot solution includes at least one of hot pregnant leach solution, hot solvent extraction raffinate, water, or other fluid.

25 32. A method as claimed in any one of claims 1 to 31 which includes determining an optimum heap configuration for a bio-assisted heap leach process of an ore heap; by measuring the leaching rate, the heat of reaction, and the sulphide content of the ore; and determining maximum aeration and irrigation rates and an
30 optimum heap height.

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33. A method as claimed in any one of the preceding claims including introduction of microorganisms into the heap of material comprising:

- a) preparing microorganisms substantially without exopolymers on their external cell walls;
- b) adding microorganisms prepared according to step a) to the heap;
- c) at least one of un-assisted or assisted re-activation of the microorganisms in the heap to produce exopolymers on their external cell walls.

34. A method as claimed in claim 33 in which step a) includes exposing the microorganisms to a low nutrient environment or starving the microorganisms.

- 35. A method as claimed in claim 34 in which the microorganisms are starved by limiting the amount of carbon available to the microorganisms.

- 36. A method as claimed in any one of claims 33 to 35 in which step b) includes one or more of adding microorganisms to the heap during formation thereof, drip irrigation of the heap, sprinkling of the heap, and pressurized irrigation of the heap.

- 37. A method as claimed in any one of claims 33 to 36 in which the assisted re-activation comprises exposing the microorganisms to a nutrient rich environment.

- 38. A method as claimed in claim 37 in which the microorganisms' environment is enriched by means of at least one of:

- a) embedding solid nutrients in the heap;

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- b) irrigating the heap with a nutrient rich solution;
- c) aerating the heap with a nutrient rich gas; and
- d) aerating the heap with a gas enriched in carbon dioxide.

5 39. A method as claimed in claim 38 in which includes the step of embedding a carbon source in the heap.

40. A method as claimed in claim 39 in which the carbon source comprises carbonate.

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41. A method as claimed in claims 38 in which the solid nutrients of step a) comprise slow release nutrients.

15 42. A method as claimed in claim 38 in which the gas of the step c) is enriched with one or more of a nutrient aerosol and ammonia.

20 43. A method as claimed in any one of claims 33 to 42 in which the un-assisted re-activation includes re-activation due to one or more of prevalent conditions in the heap and natural gas flow through the heap.

44. A method as claimed in claim 43 in which the natural gas includes carbon dioxide.

25 45. A method according to any one of claims 1 to 32 which includes the step of enriching the environment of microorganisms embedded in a heap of material for bio-assisted heap leaching by means of:

- a) embedding solid nutrients in the heap;
- 30 b) irrigating the heap with a nutrient rich solution;
- c) aerating the heap with a nutrient rich gas; and

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d) aerating the heap with a gas enriched in carbon dioxide

46. A method as claimed in claim 45 which includes embedding a carbon source in the heap.

5

47. A method as claimed in claim 46 in which the carbon source comprises carbonate.

48. A method as claimed in claims 45 in which the solid nutrients of step a) comprises slow release nutrients.

10

49. A method as claimed in claim 45 in which the gas of the step c) is enriched with one or more of a nutrient aerosol and ammonia.

50. A method as claimed in any one of claims 1 to 32 and 33 to 49 in which a sulphide fuel material is added to the heap during stacking thereof.

15

51. A method as claimed in claim 50 in which the sulphide fuel includes pyrite or other suitable sulphide concentrate.

20

52. A method as claimed in any one of the preceding claims in which irrigation is applied intermittently.

53. A method as claimed in claims 4 to 52 in which aeration is intermittently forced through the heap.

25

54. A method as claimed in any one of claims 1 to 32 in which the heap is divided into at least two zones and the process is at least partly independently controlled in each zone.

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55. A method substantially as herein described with reference to Example 1 and Figures 21 to 24.
56. A method substantially as herein described with reference to
5 Example 2 and Figures 21 and 25 to 27.
57. A method substantially as herein described with reference to Example 3 and Figures 21 and 28 to 30.
- 10 58. A method substantially as herein described with reference to Example 4 and Figure 31.

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